ELECTROFORMED MICROHARDNESS STANDARDS*

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Abstract: One of the most commonly quoted properties of a material is its microhardness. Accurate measurements of this property require the use of precise hardness standards to verify that the testing machine and testing procedure are accurate; however, it is well recognized that currently available standards vary considerably in hardness from point to point. In order to provide more uniform standards, the material used was prepared by electroforming technology. Two standards are now in production; one at 125 KHN and the second at 550 KHN. The hardness values are certified at loads of 0.245, 0.490, and 0.981 Newtons (25, 50, and 100 gram-force) with both Vickers and Knoop indentors. These electroformed materials have standard deviation in hardness, particularly at low loads, significantly better than current commercially available standards. The fabrication of the new standards and their certification procedures will be discussed.

Key Words: Hardness; knoop hardness; metrology of hardness testing; non-destructive testing; vickers hardness.

Introduction: Microhardness standards serve as a very important means of quality control, not only for electrodeposited coatings, but also for many other metallurgical applications, and can also be used to insure that the testing machines are operating properly. Presently, the only standards available are produced by the makers of the testing machines, and these lack not only a uniform standard of certification of their hardness but also exhibit a significant variation of hardness across their testing surfaces. Methods to fabricate microhardness standards that do not have the above mentioned drawbacks have been developed at the National Bureau of Standards. By electroforming microhardness standards, very close control over the three most important variables in electroplating -- current density, temperature, and electrolyte agitation is possible. By controlling these three variables, it is possible to produce extremely uniform material which can become a better certified standard. To date, two standards have been produced, one from a bright copper electrolyte and another from a bright nickel electrolyte. At present, most available

*This work was sponsored through the NBS Office of Standard Reference Materials.

standards are produced from cast alloys. The main drawback with this process is that it is extremely difficult to control cooling of the melt. This inability to cool evenly, produces variable grain structure and composition, thus producing a variable hardness across the testing surface. Copper was chosen for the first standard because the nominal hardness of 125 KHN is not only widely used but most closely represents widely tested noble metals. Nickel was chosen because the nominal hardness of 550 KHN closely represents many ferrous metals.

Electroplating, using proprietary brightners, produces grain refinement and grain distribution superior to that achieved with no brightners. This results in a significantly more uniform hardness value across the surface, as well as a bright, smooth surface. Proprietary brightners contain a leveling agent, usually an organic, which is absorbed on the surface of the high current density areas or peaks on the plating surface. This organic inhibits plating on these high current density areas, but allows plating to continue in the valleys, thereby providing a leveling effect. Another characteristic of the bright electrolytes is very good micro-throwing power, e.g., ability to deposit metal in grooves and cracks where these surface imperfections are of a microscopic nature.

<u>Fabrication Procedure</u>: The plating electrolytes used in the electroforming of microhardness standards were commercially available copper and nickel solutions. The electrolyte volume was 100 liters. The anodes were 0.004% phosphorized copper and low sulfur, low cobalt nickel bars placed in anode bags. The power supply was a 15A, 100V constant current source. The electrolyte agitation was provided by filtered air. The substrate used in this process was a 22.5 cm X 45 cm sheet of 1010 polished steel, mounted in a teflon box. Mounting the substrate in a box with the open side facing the anodes provides for a much more uniform current distribution. A uniform current density is required for uniform grain size, essential for this project. Electroforming is stopped when 1 mm of copper or nickel is deposited. Coatings of 500μm of copper on a copper substrate and on a steel substrate showed no significant difference in hardness, therefore the less expensive steel substrate was chosen. The polishing procedure removes approximately 125μm of material from the original coating of 1 mm, thus leaving a substantial coating thickness to prevent any anvil effect from the steel substrate. After the copper or nickel has been deposited, a 2.5 cm strip is removed from all four sides of the plate to ensure thickness uniformity across the plate.

The electroformed plate is cut into 1.35 cm square coupons. All cutting procedures incorporate a high speed, water cooled, carborundum cut-off wheel to minimize deformation. The coupon is then placed in a stainless steel ring 2.5 cm OD, 1.0 cm in height. This ring is then filled with an epoxy medium, used as a mold to enhance uniform polishing. The mold is polished on an automatic system with 600 grit paper, 6μm and 3μm diamond pastes, and a silica suspension to completion. The copper coupons are then flashed with 0.1μm of gold to inhibit tarnish and corrosion.

Certification Procedure: The certification of these test blocks was made in accordance with ASTM B578 and E384. The test machine load-time response was measured using a compression load cell, calibrated with certified weights, to ensure conformity to ASTM E384, Part B. The load cell was used to determine the actual load being applied to the test block during the time of indentation, as well as, determining the dwell time for full load application. The optical measuring system of the test machine was calibrated by a certified stage micrometer. The hardness indentations are made at five different areas of the test blocks and are measured using a 100x objective lens having a numerical aperature of 0.95. The hardness values are certified at loads of 0.245, 0.490, and 0.981 Newtons (25, 50, and 100 gram-force) with both Vickers and Knoop indentors.

Results: Microhardness uniformity was tested using 5 and 25 g-f loads. The 5 g-f load was chosen due to the fact that it is the most sensitive probe of surface properties and was felt to be the lowest load that could be used accurately. Loads less than 5 g-f are extremely sensitive to many variables, e.g., vibration, noise, and air currents. The results obtained from these tests were compared to two commercial standards made of brass and steel. These comparisons are listed in Table 1. Note the standard deviations for these electroformed test blocks are lower than the cast alloy test blocks, indicating improved grain size uniformity and distribution.

Discussion: Electroforming as a materials processing technique provides a substantially more uniform material with respect to hardness than is provided by the available cast alloys used in commercial standards. Hardness standards of 125 KHN and 550 KHN are being produced and are available through the NBS Office of Standard Reference Materials. The loads used are traceable to NBS fundamental mass standards, as are the stage micrometers used to calibrate the optical instrumentation used to measure the hardness indentation.

Future research in the area of microhardness will include the development of three additional standards, lead or tin-lead, with a hardness of 100 KHN or less, electroless nickel with a hardness of 900 KHN, and heat-treated cobalt-phosphorus with a hardness of 1200 KHN.

TABLE 1. A COMPARISON BETWEEN NBS STANDARDS AND COMMERCIALLY AVAILABLE STANDARDS

STANDARD	KNOOP HARDNESS (5 g-f)	
	MEAN	STANDARD DEVIATION
COPPER ON STEEL AS PLATED	131	12.2
COPPER ON STEEL, POLISHED	138	4.8
COPPER ON COPPER AS PLATED	117.2	6.18
COPPER ON COPPER, POLISHED	125.3	4.45
COPPER ON COPPER, POLISHED AND GOLD FLASHED	128.4	3.83
COMMERCIAL STANDARDS		
BRASS	119.4	6.1
STEEL (a)	1103	104
STEEL (b)	1006	87.4
NICKEL ON STEEL AS PLATED	631.3	20.4
NICKEL ON STEEL AS POLISHED	588	17.3

NOTE: THE AVERAGE THICKNESS OF THE COATING EXCEEDED 500 MICRONS

APPLICATION OF THE CHARGE DECAY NDE TECHNIQUE TO SURFACE COATINGS

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Abstract

A nondestructive evaluation technique has been recently developed for the detection of voids, inclusions, and other subsurface defects in polymeric and other insulating materials. This non-contact technique can also be used to measure the thickness of coatings on metallic substrates. Coating debonds are also detectable.

To apply this method, electrical charge is deposited onto the specimen to be tested and becomes injected into the material. This results in a surface voltage which is measured with an electrostatic voltmeter. Coating thickness variations or debonded regions lead to variations in this potential distribution.

Electrostatic charge decay nondestructive evaluation has been successfully applied in measuring epoxy coating thickness and holds much promise in many other areas.